High-Throughput Peel Measurement of a Pressure-Sensitive Adhesive

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Pressure-sensitive adhesives (PSAs) are commonplace in our everyday lives.[1-2] Surgical tape, box-sealing tape and self-stick products such as adhesive stamps and removable adhesive notes are just some of the consumer products that use pressure-sensitive adhesives. PSAs are used in space and automotive applications as well.

Pressure-sensitive adhesives are generally coated onto a backing material such as paper, cloth or polymers to support the thin adhesive layer. They adhere to surfaces with minimal applied pressure, remain tacky at room temperature, do not chemically react, and can be cleanly removed from a surface.

Because of their variety of end uses, PSAs are applied in a range of operating temperatures and conditions. No theory has been able to predict *a priori* the behavior of a PSA based on the properties of the adhesive and substrate.[3] Therefore, multiple testing must be performed. One of the most common tests is the peel test. The peel force measured is not an inherent property of the adhesive, but depends on many variables, such as the test method, temperature, peel rate, degree of contact, adhesive chemistry and thickness, aging, adhesive backing, and the substrate. Common peel tests include the T-peel test, the 180° peel test, and the 90° peel test.[4-5]

Figure 1 illustrates a 90° peel-force measurement, where the measured peel force, normalized by width of the tape, is shown as a function of peel length. The normalized peel force increases from zero to a steady peel force within 2 mm. Data from the first 2.5 cm of the peel are generally rejected, and the next 5 cm of the results are evaluated to give an average peel force.[2] We will define this average peel force measurement as the macroscopic average peel force.

Peel tests are often performed at various testing speeds and temperatures while keeping many other experimental parameters constant, such as the adhesive chemistry and thickness. The Williams, Landel and Ferry (WLF) time-temperature superposition equation enables a set of peel force curves measured at various frequencies or temperatures to be reduced to a single master curve (see Figure 2).[6-7] The peel rate increases from left to right on the graph, whereas the temperature increases from right to left. At very low temperatures (high peel rates), unsteady (shocky) peel is observed. As the temperature is increased, a region of steady (smooth) peel with clean interfacial adhesive failure occurs. For crosslinked adhesives, the peel force continues to decrease with increasing temperature. For uncrosslinked adhesives, another transition occurs at higher temperatures (low peel rates) characterized by a

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change from interfacial failure to cohesive splitting of the adhesive, leaving residue on the tape and the peel surface. The peel force often increases at this transition, followed by another region of decreasing peel force with increasing temperature. The peel force master curve identifies the best operating conditions for an adhesive. Because many peel tests are performed at various peel rates or temperatures, obtaining a peel force master curve can take a day or more. Measurement of the peel force as a function of temperature alone can provide much of the general characterization information of the master curve and allow a comparison of adhesives. The information about the relationship between temperature and peel rate contained in the shift factor is not obtained by this means, however. Often, peel forces are measured at one standard peel rate, and the adhesive performance is inferred from these results.

FASTER IS KEY: HIGH-THROUGHPUT METHODOLOGY

To alleviate the extensive testing required to produce a peel force master curve, we have applied high-throughput methodology to the peel test. High-throughput methodology challenges standard practices by developing procedures and measurement methods that can be performed faster yet give similar information. In this case, instead of measuring multiple peel tests at each desired temperature, we measured the peel force from a surface that varied linearly in temperature across the surface. As the tape is peeled, the adhesive is peeled from the surface at a different temperature. Since we can correlate temperature with peel distance, we are able to determine the temperature dependence of the peel force with one peel test.

One important parameter in designing the temperature gradient plate is determining the minimum peel length necessary to obtain an accurate peel force measurement. As previously mentioned, peel forces are averaged over a peel length of at least 5 cm in traditional peel force measurements. For these high-throughput measurements, we wish to measure an average peel force at each 1°C temperature increment. Because of the space constraints, the peel length measured at each temperature must be much smaller than the traditional 5 cm length. We define a microscopic average peel force where the data is averaged over a smaller peel length than in traditional measurements. Our goal is to obtain a microscopic peel force measurement within ± 10% of the macroscopic peel force measurement.* One might assume that the minimum peel length needed to obtain a microscopic average peel force within ± 10% of the macroscopic peel force will be the distance where adhesive deformation occurs during the peel, defined as the peel zone. It is estimated that the peel zone is comparable to the adhesive thickness, which for this experiment was 25 µm.[3] The experiment is designed to peel at least three peel zones per °C. Therefore, we desired the peel length to be at least 75 µm/°C and designed the plate to fit this requirement. The length of the peel at each particular temperature will depend on the temperature range and plate dimension. Ideally, the most information will be obtained using the largest temperature range, i.e. the steepest gradient. A steep gradient, however, gives an increasingly short peel length at each temperature.

Figure 1. Measured 90° peel force, normalized by the tape width, as a function of peel length for a commercial PSA tape (Scotch Box Sealing Tape 353). The peel force was measured at a constant temperature of 22°C on a stainless-steel plate as described in the article. The tape width was 1.9 cm and the peel rate was 1.0 mm/s. The peel force was measured to ± 5%.

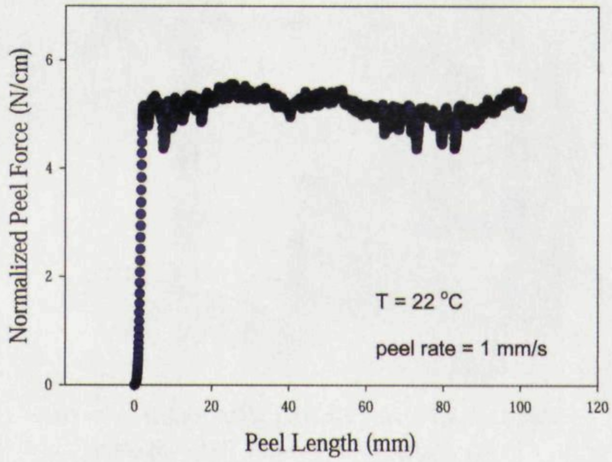
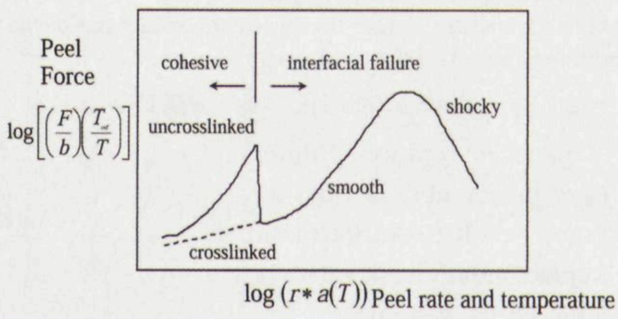


Figure 2. Typical shape of a peel force master curve for a PSA. The peel rate increases from left to right on the graph whereas the temperature increases from right to left. F is the peel force, b is the tape width, T is the temperature, r is the peel rate, and a(T) is the temperature dependent shift factor.



* Unless otherwise noted, the ± refers to the standard uncertainty in the measurements and is taken as one standard deviation of the observed values.

Figure 3. Diagram of the gradient temperature plate. The plate (A) consisted of 304 stainless steel, was 4.8 mm ± 0.1 mm thick with a #8 polished finish. The distance between the heating (F) and cooling (C) cylinders could be adjusted to change the length of the temperature gradient (via B). D shows the position of the thermocouple ports. E represents ceramic spacers to thermally insulate the heated plate from the movable stage.

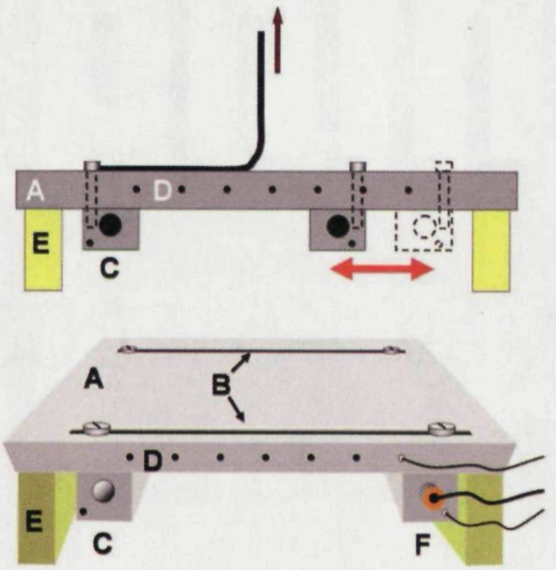
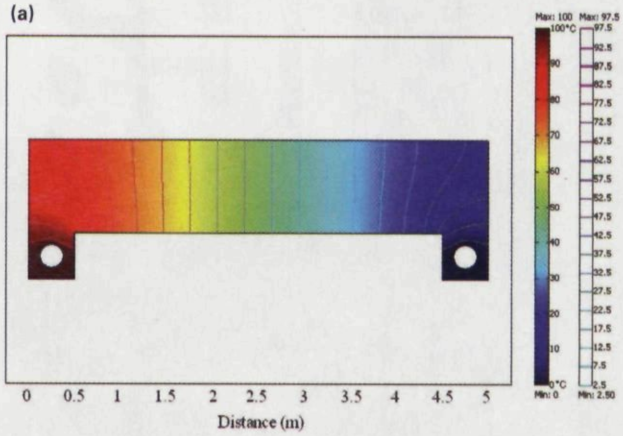
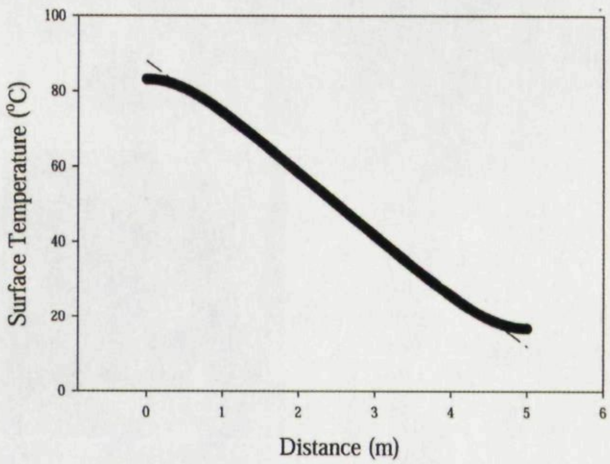


Figure 4a-b. Results of Finite Element modeling of a cross-section of the gradient temperature plate. (a) Constant temperature contour lines of the temperature of the plate. The temperatures represented by the constant contour lines are given on the far right of the graph. The heating element is set at 100°C and the cooling element is set at 0°C. (b) Temperature profile along the upper surface of the plate as a function of distance down the plate.

(a)



(b)



The gradient temperature plate was made of type 304 stainless steel and was designed as shown in Figure 3.[8] The 15 cm x 18 cm plate was 4.8 mm ± 0.1 mm thick and had a #8 polished finish. The surface roughness, Ra, corresponded to 0.2 μm. One end of the plate was attached to a cylindrical heater (Omega Engineering Inc., Stamford, CT); the other end of the plate had an open channel attached to it. Cold water was continuously run through the open channel to chill one end of the plate. Heat flows from the hot end of the plate to the cool end by way of thermal conduction.

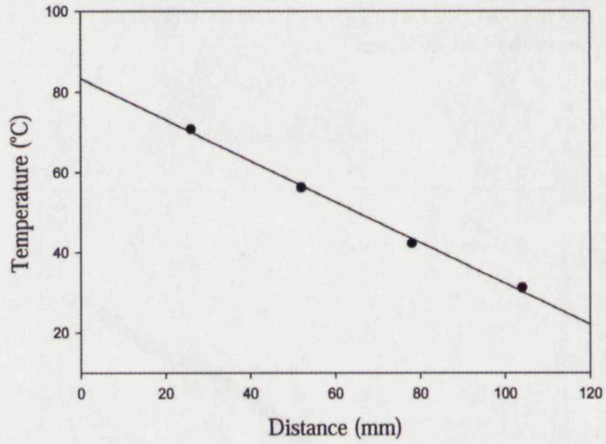
Analysis of the temperature profile in a geometry similar to the experimental gradient plate was conducted using the finite element method (FEM) with the commercial package FEMLab (FEMLab 3.0 Users Manual, COMSOL Inc., Burlington, MA [2004]). The geometry, temperature boundary conditions and solution are depicted in Figure 4a. The circular cutouts in the geometry correspond to the hot and cold channels in the gradient plate, and for the simulation, the temperature was held constant along those circular boundaries. No-flux boundary conditions were used along the rest of the structure. The solution shows that the constant temperature contour lines across the central portion of the plate are approximately equi-spaced, indicating a linear gradient. This is further confirmed by the temperature profile along the upper surface, which is plotted in Figure 4b. The plot shows that the slope in the center portion of the plate is linear, and that deviations from linearity occur only in the last 10% of each side; measurements are performed well within the linear range. Other models of the temperature gradient have included convection, and the temperature gradient is again linear.[9]

Holes were drilled in the side of the plate that allowed a thermocouple to be inserted. The temperature of the plate as a function of distance down the plate could be measured (see Figure 5). For the experimental temperature range of 50°C, the measured temperature gradient is linear as predicted from the FEM analysis. The spacing between the heater and the cooling channel could be varied to adjust the steepness of the temperature gradient. The gradient plate is similar to a Kofler hot bench used to measure melt points of organic compounds.[10-11]

The plate was bolted onto the movable stage of a texture analyzer (Texture Technologies Corp., Scarsdale, NY). The texture analyzer was configured to measure the tensile force at a 90° angle to the stage. The movable stage allows the 90° angle to be maintained as the tape is pulled from the surface.

The stainless-steel gradient plate was cleaned according to ASTM recommended cleaning procedures: wash three times with diacetone ether and dry with a tissue after each wash.[5] This was followed by washing three times with acetone, again drying with a tissue after each wash.

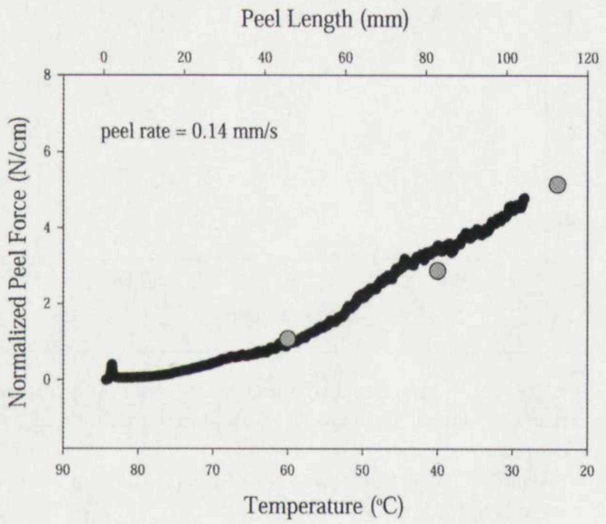
Figure 5. Calibration of the gradient temperature plate. The temperature was linear along the distance of the plate. The temperature was measured to ± 2°C.



The plate was then allowed to reach equilibrium temperature by heating one end of the plate and cooling the other end for two hours prior to testing.

After the plate had equilibrated for two hours, a 1.9 cm strip of PSA tape was applied to the gradient plate using a 2 kg rubber roller. The adhesive tape (Scotch® Box Sealing Tape 353, 3M, St. Paul, MN) is a commercially available rubber resin adhesive tape. The nominal adhesive thickness is 25 μm. The adhesive is sup-

Figure 6. Comparison of the measurements of 90° peel of Scotch® Box Sealing Tape 353 adhesive tape using the gradient temperature plate (solid line) with traditional constant temperature measurements (gray circles). The peel force was measured to ± 5% and the temperature was measured to ± 2°C. The tape width was 1.9 cm and the peel rate was 0.14 mm/s. The peel force is normalized by the width of the PSA tape.



ported on a biaxially oriented polyester backing which has a thickness of 23 μm. The "hot" end of the tape is clamped by a metal grip on the texture analyzer. The adhesive was peeled from the plate at a rate of 0.14 mm/s. The tape was peeled less than 5 minutes after the PSA tape was applied to the surface.

The solid line in Figure 6 shows results of a 90° peel measurement from the gradient temperature plate. For the particular measurement reported, the peel length was (2 ± 0.2) mm /°C. Since the adhesive thickness was 25 μm, we are peeling approximately 10 peel zones per °C. The gray data points represent traditional peel measurements taken at a constant temperature where the peel force was averaged over the length of the peel. Clearly, both measurements are giving the same peel force at a particular temperature.

The results presented in Figure 6 demonstrate that the temperature dependence of the peel force can be determined in one peel test. Multiple peel tests at different temperature ranges can be combined to give a peel force master curve over a larger temperature range. In addition, the WLF equation can be directly tested on peel measurements by comparing the peel forces measured in the high-throughput technique and those predicted by the WLF equation. Future experiments will investigate this question.

BENEFITS AND OPPORTUNITIES OF HIGH-THROUGHPUT PEEL TESTING

The results presented demonstrate that accurate peel testing can be achieved using a gradient temperature plate. The gradient temperature plate can be tailored to any temperature range or peel rate desired. Not only can peel force measurements be obtained more

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quickly, but peel transition temperatures can easily be determined.

High-throughput testing allows faster screening of adhesives, which will influence the direction and pace of product development. A number of other peel force variables can also be investigated using high-throughput methodology, including measuring the peel force as a function of a continuously varying peel rate or as a function of adhesive thickness.[12-13]

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For more information on other high-throughput measurements in materials science, visit the N.I.S.T. Combinatorial Methods Center at http://www.nist.gov/combi.

Note: Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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